ORIGINAL ARTICLE

Physical properties of self-, dual-, and light-cured direct core materials

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Abstract The objective of this study is to evaluate flexural strength, flexural modulus, compressive strength, curing temperature, curing depth, volumetric shrinkage, water sorption, and hygroscopic expansion of two self-, three dual-, and three light-curing resin-based core materials. Flexural strength and water sorption were measured according to ISO 4049, flexural modulus, compressive strength, curing temperature, and curing depth according to well-proven, literature-known methods, and the volumetric behavior was determined by the Archimedes' principle. ANOVA was calculated to find differences between the materials' properties, and correlation of water sorption and hygroscopic expansion was analysed according to Pearson ($p < 0.05$). Clearfil Photo Core demonstrated the highest flexural strength (125 ± 12 MPa) and curing depth (15.2 ± 0.1 mm) and had the highest flexural modulus ($\approx 12.6 \pm 1.2$ GPa) concertedly with Multicore HB. The best compressive strength was measured for Voco Rebilda SC and Clearfil DC Core Auto ($\approx 260 \pm 10$ MPa). Encore SuperCure Contrast had the lowest water sorption (11.8 ± 3.3 μg mm^{-3}) and hygroscopic expansion (0.0 ± 0.2 vol.%). Clearfil Photo Core and Encore SuperCure Contrast demonstrated the lowest shrinkage ($\approx 2.1 \pm 0.1$ vol.%). Water sorption and hygroscopic expansion had a very strong positive correlation. The investigated core materials significantly differed in the tested properties. The performance of the materials depended on their formulation, as well as on the respective curing process.

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Introduction

Several dental materials not specifically developed for this purpose are used for core build-up of badly broken-down teeth [1–9]. Such materials are filling resins [2, 9] (micro-hybrids, nano-hybrids, ormocers), polyalkenoate cements (glass ionomers) [2, 10], resin-modified polyalkenoate cements [10], silver-reinforced polyalkenoate cements (cermet cements) [9, 10], and the clinically well-proven amalgam [2, 9, 11]. Most of these materials provide appropriate properties as, for instance, sufficient flexural and compressive strength and flexural modulus to resist multidirectional masticatory forces. Kovarik et al. [12] reported that amalgam cores had the lowest failure rate, followed by resin composite cores, but all teeth restored with crowns over polyalkenoate cement core buildups failed. Furthermore, improvements in dentin bonding systems accompanied by a paradigm shift from "post-and-core stabilization" to "adhesively restored core retention" have stimulated the increasing use of resin-based materials for core build-up [13–15].

There are also specific resin-based core build-up materials available (Table 1), which are self-, dual-, or light-curing. Besides flexural and compressive strength or flexural modulus [5, 9, 11], other properties, such as hardness [1, 3], polymerization shrinkage [16, 17], or hygroscopic expansion [17], were also investigated for these specific products. It was found that these materials provided appropriate mechanical properties similar to amalgam and superior to cermet cements [5, 9, 11]. Their polymerization shrinkage and hygroscopic expansion was determined to be significantly lower than those of the

Table 1 Test materials

Material	Code	Formulation	Manufacturer
Clearfil Core New Bond X-ray opaque Universal Paste #02717A Catalyst Paste #02528B self-curing	CCNB	Universal Paste: Bis-GMA, TEGDMA, colloidal silica, accelerators, pigments, others Catalyst Paste: Bis-GMA, TEGDMA, colloidal silica, barium glass filler, initiators, filler content: 78 wt.%, 61 vol.%	Kuraray Europe GmbH, Frankfurt/M.,Germany
Clearfil DC Core Auto #00023A X-ray opaque dual-curing	CDCC	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, colloidal silica, barium glass filler, dl-camphorquinone, accelerators, pigments, others, filler content: 74 wt.%, 61 vol.%	Kuraray Europe GmbH
Clearfil Photo Core #02120A X-ray opaque light-curing	CPC	Bis-GMA, TEGDMA, colloidal silica, dl-camphorquinone, filler content: 83 wt.%, 68 vol.%	Kuraray Europe GmbH
Encore SuperCure Contrast #62399 X-ray opaque light-curing	ESCC	Bis-GMA, UDMA, colloidal silica, barium borosilicate glass, dl-camphorquinone, filler content: 84 wt.%, 60 vol.%	Centrix Ind., Shelton, CT, USA
Encore SuperCure natural #56724 X-ray opaque light-curing	ESCN	Bis-GMA, UDMA, colloidal silica, barium borosilicate glass, dl-camphorquinone, filler content: 84 wt.%, 60 vol.%	Centrix Ind.
Multicore HB X-ray opaque Base #H30848 Catalyst #H20610 dual-curing	MHB	Bis-GMA, TEGDMA, colloidal silica, UDMA, barium glass filler, Ba-Al-fluorosilicate glass, YbF_3, dl-camphorquinone, dibenzoyl peroxide, accelerators, pigments, others, filler content: 80 wt.%, 70 vol.%	Ivoclar Vivadent AG, Schaan, Liechtenstein
Voco Rebilda DC #600965 X-ray opaque dual-curing	VRDC	Bis-GMA, UDMA, DDDMA, silica, dl-camphorquinone, bariumborosilicate glass ceramic, dl-camphorquinone, dibenzoyl peroxide, accelerators, filler content: 71 wt.%, 57.3 vol.%	Voco GmbH, Cuxhaven, Germany
Voco Rebilda SC #601349 X-ray opaque self-curing	VRSC	Bis-GMA, UDMA, silica, bariumborosilicate glass ceramic, dibenzoyl peroxide, accelerators, filler content: 71.5 wt.%, 57.2 vol.%	Voco GmbH

Data according to manufacturer information

Bis-GMA bisphenol-A-dimethacrylate, *UDMA* urethane dimethacrylate, *TEGDMA* triethyleneglycol dimethacrylate, *DDDMA* dodecanediol dimethacrylate

cements [16, 17]. Further important material properties are curing temperature, curing depth, or water sorption. During the light-curing process, significant heat is generated by the light- and the dual-curing core build-up materials and, therefore, control of curing temperature is important to avoid damage to adjacent tissues or the still vital pulp. Some literature reports that temperature rise did not reach the critical value that can cause pulpal damage [18], but others considered control of temperature increase to be of major importance [19, 20]. Curing depth of light-curing materials is also a critical factor [21–23] because this property is essential to assure safe curing in deeper parts of the broken tooth. Moore et al. [21] measured between 55% and 70% decrease of hardness from top to bottom for 2-mm-thick specimen layers. Water sorption affects the properties of resin materials as well. High water sorption reduces flexural modulus [24, 25] and increases hygroscopic expansion [26, 27].

It was the goal of the present investigation to examine and to compare flexural strength and modulus, compressive strength, curing temperature and curing depth, polymerization shrinkage, water sorption, and hygroscopic expansion of several self-, dual-, and light-cured resin-based core materials. The null hypotheses were that (a) there is no difference between the investigated properties of the materials and (b) water sorption does not influence hygroscopic expansion.

Materials and methods

Two self-cure, three dual-cure, and three light-cure resin core materials (Table 1) were selected to investigate and to compare their material properties. Flexural strength, flexural modulus, compressive strength, curing temperature, polymerization shrinkage, and hygroscopic expansion were tested. Curing depth of the dual- and light-cure materials was also measured. Ten specimens from each material were made for each test. The light- and dual-cured specimens were removed from the molds 1 h after the curing process was completed and the self-cured specimens were removed 2 h after the mixing process. Curing was done with a quartz-tungsten halogen device (Hilux Ultra Plus, Benlioglu Dental, Ankara, Turkey) performing an irradiance of 800 ± 67 mW cm^{-2}, which was checked periodically with the Curing Light Meter (Benlioglu Dental). The 11-mm light guide was placed directly on the specimen's surface covered with a 50-μm polyester film.

Flexural strength, flexural modulus Specimens ($25 \pm 2 \times 2 \pm 0.1 \times 2 \pm 0.1$ mm) were made according to ISO 4049 [28] and cured in five 40-s steps (overlapping half the light guide's diameter) from each side (400 s in total). After 24 h of water storage at 37°, the three-point-bending

test (universal testing machine, crosshead speed of 0.75 mm min^{-1}, Model 106.L, Test GmbH, Erkrath, Germany) was conducted. Flexural strength was calculated by $\sigma = (3FL)/(2bh^2)$ and flexural modulus by $E = (L^3/4bh^3) \times (F/Y)$, both expressed in megapascals, with F = maximum strength, L = distance between the rests (20 mm), b = width of the specimen, h = height of the specimen, and F/Y = slope of the linear part of the stress–strain curve.

Compressive strength Cylindrical specimens (length: 6 ± 0.1 mm, diameter: 4 ± 0.1 mm) were cured for 40 s from each side and crushed between two parallel plates after 24 h of water storage at 37° (universal testing machine, crosshead speed of 0.75 mm min^{-1}). Compressive strength was calculated by $S = F/\left((d/2)^2 \times \pi\right)$ expressed in megapascals with F = maximum strength and d = diameter of the specimen.

Curing depth Dual- and light-curing materials were filled in a white polyoxymethylen mold (length: 15 ± 1 mm, diameter: 4 ± 0.1 mm) and polymerized for 40 s from one side. Immediately after irradiation and removal from the mold, the unpolymerized parts were scraped off and the length of the cured material was measured with a mechanical caliper (Special Caliper, accuracy of 0.02 mm, MIB Messzeuge GmbH, Spangenberg, Germany).

Curing temperature The mold used for the curing depth test was filled again with each material and a thermocouple wire (1.3 mm diameter) of a digital resistance thermometer (P600 Series, Dostmann Electronic GmbH, Wertheim-Reicholzheim, Germany) was centrally placed in the mold in such a way that its stripped ends (3 mm length) were level with the material's surface to be irradiated. Since light-induced polymerization starts first at the surface, this position was found to be the warmest. Dual- and light-cure materials were polymerized for 40 s from one side. During the 40-s curing cycle, the maximum temperature was recorded.

Polymerization shrinkage Polymerization shrinkage was calculated from the densities measured according to the Archimedes' principle with the commercial Density Determination Kit of the analytical balance Mettler Toledo XS (Mettler Toledo GmbH, Greifensee, Switzerland). The specimens were weighed in air and in water, and the density was calculated in grams per cubic centimeter by the software of the Mettler Toledo XS balance by $D = (A/(A-B)) \times (D_0 - D_L) + D_L$ with D = density of sample, A = weight of sample in air, B = weight of sample in water, D_0 = density of water at the exactly measured temperature in degrees Celsius according to the

density table of distilled water, and D_L = air density (0.0012 gcm^{-3}). An internal balance correction factor (0.99985) took air buoyancy of the adjustment weight into account.

From each uncured material, spherical specimens, each of approximately 0.1 g, were carefully formed so that trapped air bubbles were avoided. Each specimen was put on a polyester film (thickness 0.05), fixed on the special holder of the balance, of which the masses in air and in water were known and the masses of the whole assembly in air and in water were weighed. Since the weighing process was very fast (approximately 10 s), there was no water uptake or flow of the material. It was observed that the uncured specimens were optimally wetted. The mass of each specimen was calculated by subtracting the mass of the polyester film from the mass of the whole assembly, and the density of the uncured material (D_{un}) was computed. Next, ten discs (diameter 10 ± 0.1 mm, thickness 1 ± 0.1 mm) of each material were prepared and polymerized for 40 s from each side. Then, the masses in air, m_1, and in water and the densities (D_1) were evaluated and the polymerization shrinkage in percent was calculated by $\Delta V = ((1/D_1) - (1/D_{un})) \times (1/D_{un}) \times 100$.

Hygroscopic expansion and water sorption Cylindrical specimens of each material (diameter 10 ± 0.1 mm, thickness 1 ± 0.1 mm) were polymerized for 40 s on each side. The volume V_1 was calculated after 15 min of dry and dark storage at room temperature by measuring their diameters and thicknesses with a mechanical caliper to avoid any moisture influence (volumes of actually dry specimens). Then, mass m_1 and density D_1 of each specimen were determined. After 30 days, dark storage in water at $37\pm 1°C$ the masses m_2 and densities D_2 were measured again, and the respective volumes were calculated by $V_2=m_2/D_2$. The hygroscopic expansion ΔV was calculated by subtracting V_2 from V_1, and the results were expressed in percent. Water sorption W_{Sp} was calculated by $W_{Sp}=(m_2-m_1)/V$. Prior to each measurement, the specimens were tempered to room temperature in a water bath for 10 min. Before weighing the specimens in the air, they were blot-dried with a cellulose pad.

Statistical analysis Means and standard deviations were calculated. Normal distribution was tested by the Kolmogoroff–Smirnoff test. Univariate ANOVA and post hoc Scheffé's test were performed separately for each of the different properties and were also calculated to identify differences among the various properties of self-, dual-, and light-cured material groups (SPSS 15.0, SPSS, Chicago, IL, USA). T test for unpaired samples was calculated to find differences between the light- and the dual-cured material groups for curing depth. Correlation

analyses were calculated according to Pearson. Statistical significance for all tests was considered as $p<0.05$.

Results

The results are given in Table 2. Considering the differently cured material groups, it was found that flexural strength of the dual-cured material group significantly differed from the light-cured group ($p=0.013$). Flexural strength positively correlated with volume percent filler content (0.231, $p<0.034$) but did not correlate with weight percent filler content. Flexural moduli of the light-cured ($p=0.001$) and the dual-cured material groups ($p=0.006$) were significantly higher than those of the self-cured material group. Flexural modulus positively correlated with volume percent (0.779, $p<0.000$), as well as with weight percent (0.663, $p<0.000$) filler content, while compressive strength negatively correlated with volume percent (−0.496, $p<0.000$) and weight percent (−0.731, $p<0.000$) filler content. Compressive strength of the light-cured material group significantly differed from the self-cured ($p=0.006$), but no difference was found between the self- and dual-cured groups. All materials significantly differed from each other for curing temperature ($p=0.000$), which increased from the self- to the dual- and to the light-cured material group. Curing depth of the light-cured materials was significantly higher ($p<0.00$) than of the dual-cured materials. Polymerization shrinkage of the light-cured material group was significantly lower than that of the dual-cured ($p=0.002$) and self-cured ($p=0.044$) groups. No correlation of polymerization shrinkage and filler content was found. No differences between the differently cured material groups were found for water sorption and hygroscopic expansion. Water sorption and hygroscopic expansion showed positive and highly significant correlation (0.615, $p=0.000$).

Clearfil Photo Core (CPC) demonstrated the highest flexural strength and curing depth and, concertedly with Multicore HB (MHB), had the highest flexural modulus. The best values for compressive strength were measured for Voco Rebilda SC (VRSC) and Clearfil DC Core Auto (CDCC). Encore SuperCure Contrast (ESCC) had the lowest water sorption and hygroscopic expansion. CPC and ESCC showed the lowest shrinkage. Although not significant, filler volume and shrinkage tended towards positive correlation values (0.200, $p<0.087$).

Discussion

The applied methods were appropriate for evaluating the investigated properties. Flexural strength and water sorption were measured according to the dental standard ISO 4049 [28], flexural modulus was calculated from the three-point-bending test as described by the literature [29, 30], and polymerization shrinkage and hygroscopic expansion were determined by the well-proven Archimedes' principle [17, 26, 31]. Curing depth and temperature were measured in accordance with ISO 4049. The selected materials were Bis-GMA-based microhybrids differing in formulation and polymerization process. From one light-curing product, the shaded and unshaded versions were investigated (Encore SuperCure Contrast/Encore SuperCure Natural) to evaluate whether the shade influenced the properties.

All test materials highly significantly exceeded the minimum value of 50 MPa for compressive strength required for amalgamation, which is clinically well-proven for core build-ups, while many materials exceeded 180 MPa, which has been reported for the high-copper admixed silver amalgam Duralloy [2]. In this connection, it must be mentioned that other authors reported values of approximately 480 MPa for the same amalgam [9], which

Table 2 Means and (standard deviations) of the physical properties of resin-based self-, light-, and dual-cured build-up materials

	Material	Flexural strength [MPa]	Flexural modulus [GPa]	Compress. strength [MPa]	Curing temperature [°C]	Curing depth [mm]	Shrinkage [vol.%]	Water sorption [$\mu g\,mm^{-3}$]	Hygros. expansion [vol.%]
Self-cured	CCNB	107 (9)$_{14}$	8.66 (1.03)$_1$	220 (30)$_1$	29.4 (0.6)$_1$	–	−5.5 (0.8)$_1$	15.7 (3.9)$_{12}$	0.6 (0.2)$_1$
	VRSC	57 (8)$_{23}$	2.96 (0.42)	263 (12)$_{23}$	31.0 (0.7)$_{16}$	–	−6.0 (0.6)$_1$	14.9 (1.7)$_1$	0.6 (0.2)$_1$
Dual-cured	CDCC	107 (14)$_{14}$	7.58 (0.35)$_1$	268 (10)$_2$	31.0 (0.8)$_{15}$	7.8 (0.5)	−5.8 (1.3)$_1$	15.5 (2.8)$_1$	0.4 (0.2)$_1$
	MHB	95 (8)$_4$	12.55 (1.36)$_2$	116 (23)	32.1 (0.7)$_{456}$	9.7 (1.4)$_2$	−7.0 (2.6)$_{13}$	13.5 (1.6)$_1$	0.5 (0.3)$_1$
	VRDC	102 (15)$_{14}$	5.61 (0.78)	240 (28)$_{12}$	34.5 (1.7)$_2$	9.2 (1.0)$_2$	−6.1 (1.5)$_1$	15.7 (2.9)$_1$	0.6 (0.3)$_1$
Light-cured	CPC	125 (12)$_1$	12.79 (1.17)$_2$	234 (14)$_{13}$	35.2 (0.6)$_{23}$	15.2 (0.1)	−2.0 (0.2)$_2$	18.4 (3.9)$_2$	0.7 (0.2)$_1$
	ESCC	57 (3)$_2$	7.28 (0.50)$_1$	158 (22)$_4$	35.3 (1.7)$_3$	13.0 (0.8)$_1$	−2.1 (0.1)$_2$	11.8 (3.3)$_1$	0.0 (0.2)
	ESCN	73 (8)$_3$	8.12 (0.82)$_1$	169 (23)$_4$	33.1 (1.8)$_{24}$	13.9 (0.5)$_1$	−7.8 (2.1)$_3$	12.1 (1.4)$_1$	0.5 (0.1)$_1$

Data with the same subscript number within each column indicate not significant differences ($p<0.05$)

was not attained by any of the test materials. Some authors consider compressive strength to be the critical factor of success because a high compressive strength is necessary to resist masticatory and parafunctional forces [2, 32]. However, it should be observed that actually the tensile stress induces problems in brittle materials, less the compression. The present study showed compressive strength significantly depending on the activation mode. All self-cured materials, but only two dual-cured materials and only one light-cured material, demonstrated a compressive strength above 200 MPa. Furthermore, the detected negative and highly significant correlation with weight percent and volume percent filler content proved that the formulation also significantly influenced compressive strength. The values for the correlation coefficient indicated that not only the amount but also the type and size of the filler needed to be considered, which supported the literature reporting the significant effect of filler type, filler content, and particle size on compressive strength [33–35].

Core build-up materials should have high flexural modulus similar to that of tooth structure (dentin) to withstand the forces of mastication and polymerization shrinkage stresses [9]. If the modulus mismatch between the restoration and the hard tooth tissues is too high, interfacial stress may result from either thermal, mechanical, or shrinkage strain in the material [36]. Flexural modulus significantly increased from the self-cured via the dual-cured to the light-cured group. While dual- and light-cured materials were optimally polymerized, this was possibly not the case for the self-cured products because they were cured at room temperature and their polymerization process is temperature-dependent. However, it is doubted that the activation mode was the real cause for these findings since no influence on flexural strength was found. This is in accordance with the literature [37], negating an effect of activation mode on flexural modulus. Due to the found positive and highly significant correlation of flexural strength and modulus and in agreement with the literature [29, 37], filler type and content might be considered as important factors. However, the low correlation coefficient also indicated that these properties were also influenced by the chemistry of the matrix. Combe et al. [9] measured flexural modulus of approximately 17 GPa for the high-copper admixed silver amalgam Duralloy, which is significantly higher when compared with the test materials. Nevertheless, Multicore HB (MHB) and Clearfil Photo Core (CPC) performed very high moduli of approximately 13 GPa. These results also supported the assumption that the materials' formulation essentially determined flexural modulus.

Curing temperature is an important factor to be considered because tissues adjacent to the restored tooth or a still vital pulp might be damaged [18–20]. All self-cured materials generated significantly lower polymerization temperatures than the light-cured ones. This is explained by the lower speed of reaction in the self-curing polymerization process, which took several minutes compared to the light-curing process that is completed within seconds. The dual-cured material Clearfil DC Core Auto (CDCC) generated a similar level of heat to the self-cured materials but significantly less than the light-cured materials, which might be explained by smaller amounts of camphorqhinone, which is used as photoinitiator in all materials (Table 1) and, therefore, by lower speed of reaction, too. The fact that all dual-cured test materials performed significantly less curing depth than the light-cured products might also be due to smaller photoinitiator concentration. This is certainly acceptable since the self-curing process of the dual-curing materials secures safe polymerization in deep parts of the cavity.

Polymerization shrinkage depends on various factors, such as on the formulation of the resin matrix, the filler type and content, and the degree of conversion [26, 38]. The light-curing materials Clearfil Photo Core (CPC) and Encore SuperCure Contrast (ESCC) had the lowest polymerization shrinkage. Since no significant correlation of shrinkage and filler volume was found and the volumetric filler content of ESCC was even lower than that of CPC and not above the other materials, it was assumed that other factors, like filler size or type and resin chemistry, may also effect shrinkage [26, 39]. No explanation was found for the significant difference in shrinkage between ESCC and Encore SuperCure Natural (ESCN), which only differed in their shade.

Statistical analysis proved correlation of water sorption with hygroscopic expansion, which is in accordance with the literature [26, 40]. The high water sorption of Clearfil Photo Core (CPC) was assumed to be due to larger amounts of the hydrophilic monomer TEGDMA compared to the other test materials [25]. The very low hygroscopic expansion of Encore SuperCure Contrast (ESCC) was also explained by the lower degree of conversion of the organic matrix, implicating an exchange of residual monomer by water molecules and, therefore, resulting in hidden expansion. Hygroscopic expansion of the test materials was in accordance with the literature that also reported lower expansion of resin-based core materials than of cements [17].

After having reviewed the relevant literature from 1991 to 2003, Larson [41] concluded amalgam to be the strongest material, best able to withstand adverse stress and to restore teeth having the greatest loss of tooth structure. In his opinion, resin-based materials, whether chemically cured or light cured, reinforced or not, appeared best capable of core restoration for moderately broken

down teeth. Based on the results of the present investigation and more recent publications [2, 42], it is the authors' opinion that there are resin-based core materials with strengths similar to amalgams. Furthermore, the philosophy of achieving high retention at the core–tooth interface can be implemented much better with resin-based materials. However, no general recommendation can be given as to whether self-cure, dual-cure, or light-cure materials should be used, as the individual formulation of the material seems to be the most important parameter.

One major limitation of the present study was that no detailed information of the test materials' formulations was available. Therefore, some effects might be over-estimated and others might be underestimated. However, the obtained results of the various investigated products provide important information for characterizing resin-based core materials.

Conclusion

Within the limitations of this study, it can be concluded that the investigated core materials significantly differed in the tested properties and that their performance depended on their formulation, as well as on the respective curing process. Water sorption and hygroscopic expansion positively corre-lated. Therefore, both parts of the null hypothesis were rejected.

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